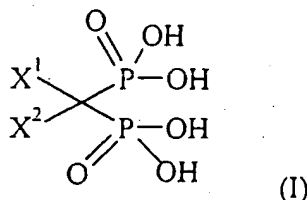
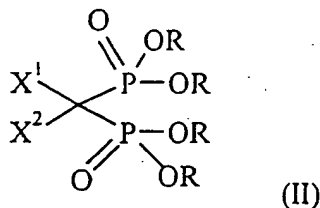


CLAIMS:

1. A process for preparing salts of a substituted or unsubstituted methylene bisphosphonic acid of general formula I



wherein X¹ and X² are independently hydrogen or halogen, which process comprises hydrolysing, using hydrochloric acid, the corresponding ester of formula II,



wherein X¹ and X² are defined above and R is a C₁₋₄ straight or branched chain alkyl group and converting the acid to a salt by reaction with a base characterised in that the
15 concentration of hydrochloric acid is from 15% to 20% by weight and water is removed azeotropically from the resultant acid using n-butanol prior to the addition of an amine or a base.

2. The process as claimed in claim 1 wherein X¹ and X² are both fluorine, chlorine or
20 bromine.
3. The process as claimed in claim 2 wherein X¹ and X² are both chlorine.

4. The process as claimed in claims 1 to 3 wherein the tetraester of formula II is the tetraisopropyl ester.

5. The process as claimed in claim 1 wherein the compound of formula II is
5 dichloromethylene bisphosphonate tetraisopropyl ester.

6. The process as claimed in any one of claims 1 to 5 wherein the acid product of the hydrolysis is reacted with an organic or inorganic base such as a C_{1-6} straight or branched primary, secondary or tertiary alkylamine, aralkylamine, basic N-containing heterocycle,
10 alkali or alkaline earth metal hydroxide.

7. The process as claimed in claim 6 wherein the base is selected from the group comprising C_{1-4} straight or branched primary, secondary or tertiary alkylamine, aralkylamine, basic N-containing heterocycle or alkali metal hydroxides.

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8. The process as claimed in claim 7, wherein the base is selected from the group comprising triethylamine, tri-n-propylamine, diisopropylethylamine, tri-n-butylamine, pyridine, tribenzylamine and sodium hydroxide.

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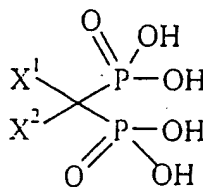
9. The process as claimed in claim 8, wherein the volume of hydrochloric acid used is from about 3 to about 5 volumes.

10. The process as claimed in any one of claims 1 to 9 wherein the hydrolysis is carried out at from about 80°C to about 90°C.

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11. The process as claimed in any one of claims 1 to 10 wherein a vacuum is applied following the addition of n-butanol.

12. A salt of substituted or unsubstituted methylene bisphosphonic acid of formula I



wherein X^1 and X^2 are independently hydrogen or halogen.

13. A salt as claimed in claim 12 wherein X^1 and X^2 are both hydrogen, fluorine, chlorine or bromine.

14. A salt as claimed in claim 13 wherein X^1 and X^2 are both chlorine.

15. A salt as claimed in claim 12 wherein one of X^1 and X^2 is hydrogen and one is chlorine.

16. A salt as claimed in any one of claims 12 to 15 wherein the salt is a C_{1-6} straight or branched primary, secondary or tertiary alkyl amine salt, an aralkyl amine salt, a basic N-containing heterocycle salt, an alkali or alkaline earth metal salt.

17. A salt as claimed in claim 16 wherein the salt is a C_{1-4} straight or branched primary, secondary or tertiary alkylamine salt, an aralkyl amine salt, a basic N-containing heterocycle salt or an alkali metal salt such as sodium or potassium salt,

18. A salt as claimed in claim 17 wherein the salt is a triethylamine, tri-n-propylamine, diisopropylethylamine, tri-n-butylamine, pyridine, tribenzylamine or sodium salt.

19. A salt as claimed in claim 18 which is selected from

dichloromethylene bisphosphonic acid, mono(tri-n-butylamine) salt

dichloromethylene bisphosphonic acid, monopyridine salt

dichloromethylene bisphosphonic acid, mono(triethylamine) salt

dichloromethylene bisphosphonic acid, mono(diisopropylethylamine) salt

dichloromethylene bisphosphonic acid, mono(tribenzylamine) salt

5 dichloromethylene bisphosphonic acid, mono(tri-n-propylamine) salt

dichloromethylene bisphosphonic acid, disodium salt.